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Achieving Large-area Bulk Ultrafine Grained Cu via Submerged Multiple-pass Friction Stir Processing

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Large-area bulk ultrafine grained (UFG) pure Cu was successfully prepared by multiple-pass overlapping friction stir processing (FSP) under additional rapid cooling. Overlapping FSP did not exert a significant effect on the microstructure and mechanical properties of the FSP UFG Cu. Similar average grain size was achieved in the transitional zone (TZ) of the multiple-pass FSP sample compared to that in the nugget zone of the single-pass FSP sample, and the TZ exhibited a strong {111}⟨112⟩ type A fiber shear texture. Very weak softening occurred in the TZ of the multiple-pass FSP UFG Cu, resulting in a relatively uniform hardness distribution throughout the whole processed zone. A high yield strength of ~310 MPa and a uniform elongation of ~13% were achieved in the bulk FSP UFG Cu. This study provides an effective strategy to prepare large-area bulk UFG materials.

KEY WORDS: Friction stir processing; Multiple-pass; Ultrafine grains; Microstructure; Mechanical properties

1. Introduction

Over the past two decades, much attention has been paid to the ultrafine grained (UFG) and nanostructured (NS) materials due to their enhanced strength and hardness[1]. Disappointingly, these materials usually exhibit very low tensile ductility at ambient temperature, which can be attributed to the artifacts from processing, the plastic instability with little or no strain hardening (dislocation storage) capacity, and low resistance to crack initiation and propagation[2].

Various severe plastic deformation (SPD) methods, such as equal channel angular pressing (ECAP), high pressure torsion (HPT), dynamic plastic deformation (DPD), provide practical approaches to produce 100% dense UFG materials that can exhibit mechanical properties controlled by their intrinsic deformation mechanisms[1,3]. However, these UFG materials also exhibit low tensile ductility owing to their insufficient strain hardening capacity, and the uniform elongation is usually very low or near zero[4]. Therefore, preparing UFG materials with enhanced strength and ductility by simple processing methods is still an important challenge.

Friction stir welding (FSW), which is a solid-state welding process that involves severe plastic deformation, has been widely used in joining of light alloys and ferrous alloys[5–11]. Based on the basic principles of FSW, a new processing technique – friction stir processing (FSP) has been developed for microstructural modification[5]. FSP is an effective method for producing fine-grained structure and surface composite, modifying the microstructure of materials, and synthesizing the composite and intermetallic compound in situ[12]. Recently, FSP has been successfully used for producing UFG microstructures in Al, Mg, Cu alloys and steel[13–17].

FSP UFG materials usually exhibited equiaxed recrystallized grains with relatively low dislocation densities, and the fraction of high angle grain boundaries (HAGBs) is very high (~90%)[15,16,18]. Moreover, abundant twin boundaries can be introduced into the ultrafine grains in the FSP Cu–Al alloys with low stacking fault energies, and a dual-phase structure can be achieved in FSP UFG low carbon steel[16,17]. These studies indicated that enhanced strain hardening was achieved in these special UFG microstructures during tension, resulting in sound strength–ductility synergies[15–17].

The development of UFG materials for structural applications must address issues related to the fabrication of bulk samples. Most SPD techniques produce relatively small quantities of material which are difficult to scale up, and are unlikely to produce materials at a low cost. By contrast, FSP is able to produce large-area bulk materials via multiple-pass overlapping method[19–21]. Su et al.[20] have successfully prepared bulk UFG 7075 Al alloy by running multiple-pass overlapping FSP under

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additional cooling, and relatively uniform microstructure was obtained in the processed zone. However, they did not perform the investigations on the mechanical properties of the bulk UFG Al alloy.

Usually, a transitional zone (TZ) exists between two adjacent nugget zones (NZs) in the multiple-pass overlapping FSP sample, which may consist of the thermo-mechanically affected zone (TMAZ) and the heat affected zone (HAZ) of the later-pass processed zone. The microstructure of the TZ may be different from the NZ, and the global mechanical properties of the bulk UFG material should be influenced by the TZ. Therefore, it is essential to investigate the microstructure and mechanical properties of the bulk UFG materials prepared by multiple-pass overlapping FSP. In this study, 5-pass overlapping FSP was performed on a commercially pure copper plate. The aim is to understand the effect of the TZ on the microstructural evolution and mechanical properties of FSP bulk materials, and obtain large-area bulk UFG material with good strength and ductility.

2. Experimental

Commercially pure Cu (∼99.9%) plate, 5 mm in thickness, 300 mm in length and 100 mm in width, was used in this study, and the rotation tool was made of heat-treated tool steel (M42). Two different FSP schemes were followed: single-pass, and 5-pass with half of the pin diameter overlapping, as schematically shown in Fig. 1. For the FSP process, the pure Cu plate was first fixed in water, and additional cooling by the flowing water was adopted during FSP process. FSP was performed at a rotation rate of 400 r/min and a traverse speed of 50 mm/min, using a tool with a shoulder of 20 mm in diameter and a cylindrical threaded pin of 6 mm in diameter and 2.7 mm in length.

Microstructural observation and analysis were conducted by optical microscopy (OM) and electron backscatter diffraction (EBSD). The metallographic specimens were cross-sectioned perpendicular to the FSP direction, polished and then etched with a solution of 100 ml distilled water, 15 ml hydrochloric acid and 2.5 g iron (III) chloride. The EBSD specimens were machined on the cross-sectional plane, mechanically ground and polished, and electrolytically polished at an electric voltage of 5 V in a solution of 2.5 g carbamide, 25 ml isopropyl alcohol, 125 ml phosphoric acid, 125 ml ethanol and 250 ml water. EBSD scans were performed using an Oxford HKL Channel 5 system on a LEO Supra 35 FEG scanning electron microscope.

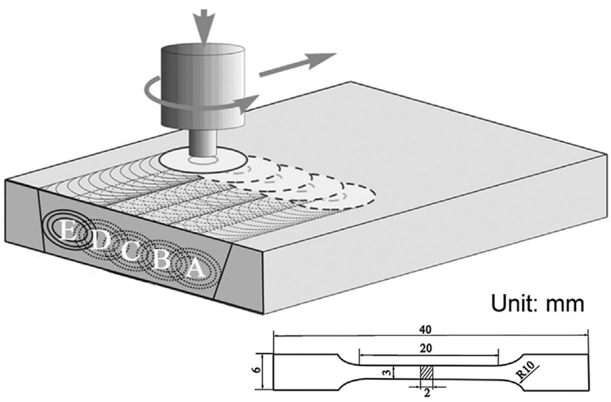

Fig. 1 Schematic illustrations of the 5-pass overlapping FSP process and the tensile specimen.

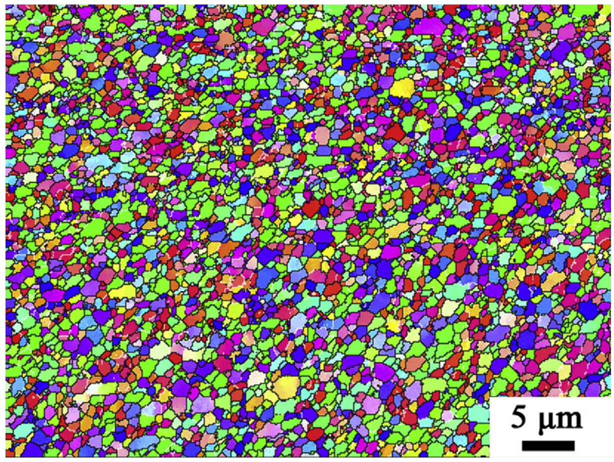

Fig. 2 EBSD microstructure in the nugget zone of the single-pass FSP sample.

The microhardness of the bulk FSP pure Cu sample was measured along the middle-thickness of the processed zone with a 50 g load for 10 s. Large dog-bone-shaped tensile specimens with a gauge length of 20 mm, a gauge width of 3 mm and a gauge thickness of 2 mm were machined perpendicular to the FSP direction from the processed zone in the bulk sample, and the tensile specimen is schematically shown in Fig. 1. Furthermore, mini tensile specimens with a gauge length of 5 mm, a gauge width of 1.4 mm and a gauge thickness of 0.6 mm were machined along the FSP direction from the NZ of the single-pass FSP sample. All the tensile tests were conducted at an initial strain rate of 1×10^{-3} s^{-1}.

3. Results and Discussion

Fig. 2 shows the typical EBSD microstructure of the UFG pure Cu from the NZ of the single-pass FSP sample. It is clear that a relatively uniform microstructure with ultrafine equiaxed grains was achieved in the FSP UFG pure Cu, indicating that dynamic recrystallization (DRX) occurred during the FSP process[5,15]. The final structure in the FSP UFG Cu sample should consist of the coarsened grains after the growth stage during DRX process. It is suggested that once a given volume of plastically deformed material was outside the deformation zone of the rotating tool pin, it coarsened very quickly under the influence of heat[22]. The average grain (including sub-grains) size was about 730 nm measured by the mean linear intercept method.

Fig. 3 shows the cross-sectional macrograph of the 5-pass overlapping FSP large-area bulk sample. The remnant regions of the NZs from the 1st to the 4th pass FSP are labeled by A to D, respectively, and the NZ of the 5th pass FSP is labeled by E. It is apparent that the processed zone exhibited a high degree of continuity and no processing defects, such as porosity or holes

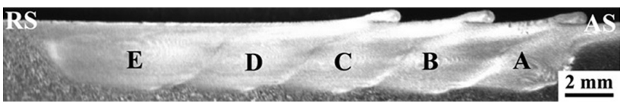

Fig. 3 Cross-sectional macrostructure of the 5-pass overlapping bulk FSP UFG Cu.

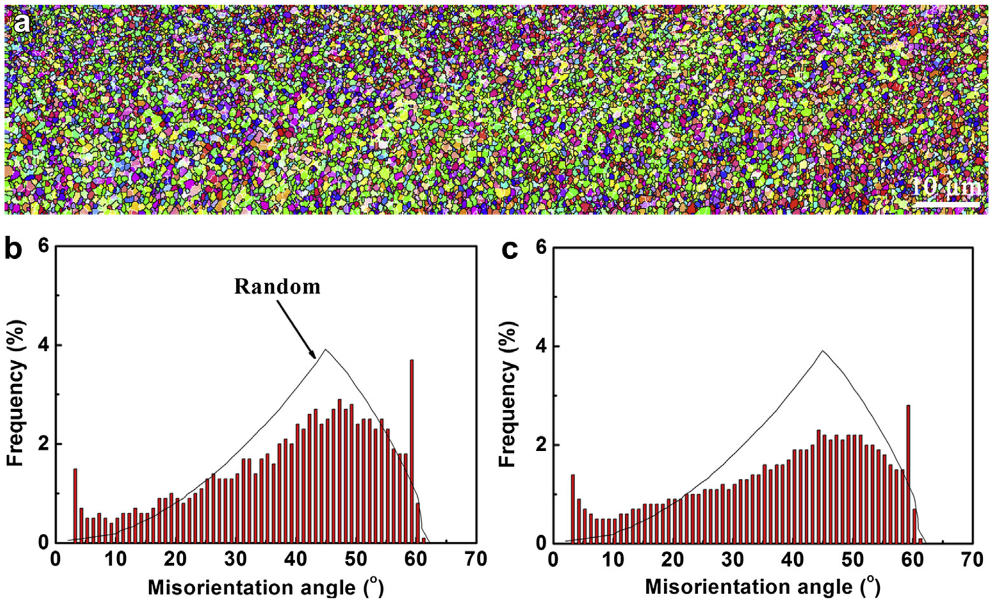

Fig. 4 (a) EBSD microstructure in the TZ, and the grain boundary misorientation distributions of (b) NZ and (c) TZ.

were detected. This indicates that high quality large-area bulk UFG pure Cu can be produced via multiple-pass overlapping FSP.

It is clear from Fig. 3 that obvious TZs can be observed between each two adjacent processed zones, and the typical microstructure of the TZ between the 1st and the 2nd passes is shown in Fig. 4(a). It can be found from Figs. 2 and 4(a) that the UFG microstructure in the TZ was similar to that in the NZ. The microstructure of the TZ also exhibited ultrafine equiaxed grains, and the average grain size was measured to be about 700 nm, which was similar to that in the NZ. This indicates that the coarsening of the UFG microstructure in the TZ was very little under the thermal and mechanical actions of the subsequent pass FSP. Moreover, the FSP UFG microstructure had sound thermal stability under the FSP condition used in this study.

Similar distributions of the grain boundary misorientation angles were obtained in the NZ and the TZ, which exhibited a nearly random distribution for a cubic polycrystalline, as shown in Fig. 4(b) and (c). Considering all grain boundaries with misorientation angles >2°, the HAGBs (misorientation angle >15°) comprised about 92% and 90% in the NZ and the TZ, respectively. Clearly, the fraction of HAGBs in the FSP UFG Cu is obviously larger than that in SPD UFG Cu samples in which the fraction of HAGBs reached only ∼60% even after complex procedures[23−25].

Fig. 5 shows the typical {111} pole figures of the NZ and the TZ, respectively. Very weak texture was achieved in the NZ with the maximum pole density value of only 2.14; however, an obvious shear deformation texture of {111}⟨112⟩ type A fiber texture was observed in the TZ with the maximum pole density value of 8.83.

FSW/P process involves significant shearing of the material around the rotating pin, so all textures in the FSW/P materials can be based upon the expected shear textures in fcc materials[26,27]. In general, shear deformation occurs easily by glide on {111} plane in ⟨110⟩ type directions. This results in the formation of two partial fiber textures during shear deformation, which derives from the {111} shear plane (A fiber) and ⟨110⟩ shear direction (B fiber), respectively. Using Miller index

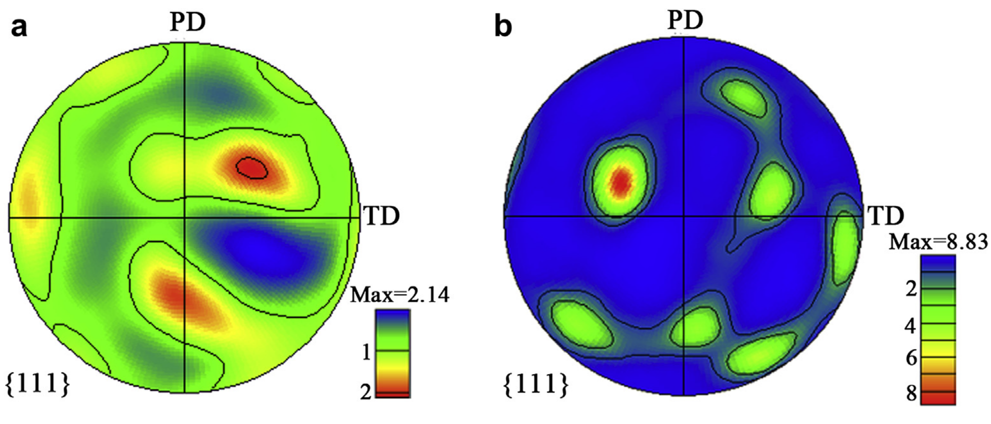

Fig. 5 Typical {111} pole figures of (a) NZ, and (b) TZ (TD: transverse direction, PD: processing direction).

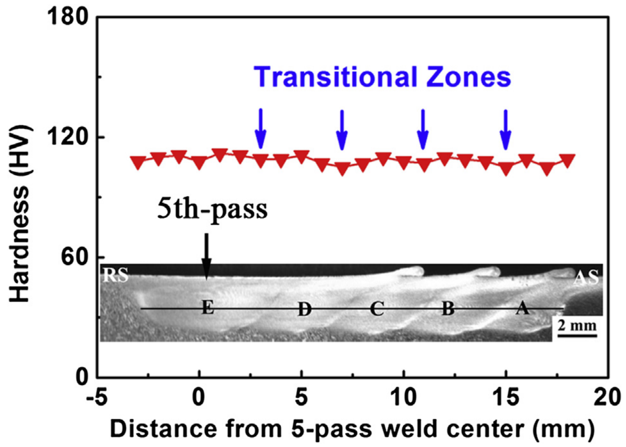

Fig. 6 Hardness distribution of the 5-pass FSP bulk UFG Cu.

notation, A fiber can be denoted {111}⟨*uvw*⟩, where the pole indicates the shearing plane, and the direction indicates the shear direction of the imposed deformation.

In fact, the TZ in the multiple-pass FSP sample is the TMAZ (or including HAZ) of the later-pass processed zone, therefore strong shearing deformation will occur in this zone. For the TZ in this study, the shear deformation preferred to rotate the crystallite lattice so that the plane of easy glide {111} lay in the shearing plane, and the shear direction was ⟨112⟩, resulting in the {111}⟨112⟩ type A fiber texture.

From the hardness line profile in the middle-thickness of the processed zone shown in Fig. 6, it is clear that relatively uniform hardness distribution was achieved throughout the whole processed zone. The lowest hardness value in the TZ was determined to be ∼105 HV which decreased slightly compared to that of the NZ with the average hardness value of ∼110 HV.

Fig. 7 shows the typical tensile curves of the multiple-pass bulk FSP sample and the single-pass FSP sample. The single-pass FSP sample exhibited a yield point elongation with a high yield strength (YS) of ∼330 MPa, and the ultimate tensile strength (UTS) and uniform elongation (UE) was ∼340 MPa and ∼18%, respectively. For the bulk sample, an obvious yield point elongation was also observed with a slightly reduced YS of ∼310 MPa and a similar UTS of ∼340 MPa. Compared to the single-pass FSP sample, the bulk FSP sample exhibited a decreased UE of ∼13%, which should be related to the texture distribution. During tension, local shear deformation may occur preferentially in the TZ with stronger shear texture and therefore result in the reduced UE. Though the UE of bulk FSP sample decreased compared to the single-pass FSP sample, the achieved UE of ∼13% is better than that for most SPD UFG Cu samples[1,2].

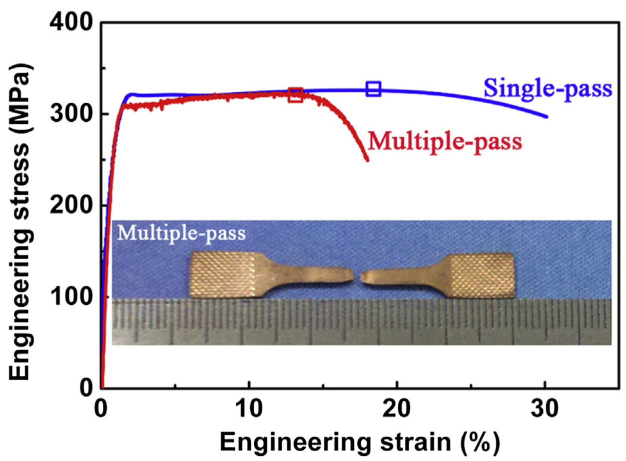

Fig. 7 Typical tensile curves of the multiple-pass FSP UFG sample (large-sized specimen) and the single-pass FSP sample (mini-specimen).

The above results indicate that very weak softening effect occurred in the TZ, and this is in accord with the microstructure observations of the NZ and the TZ in Figs. 2 and 4(a). Our previous study[28] indicated that the FSW joint of work-hardened (H state) pure Cu with nearly equal strength to the parent metal could be obtained under additional water cooling. Significantly reduced heat input with a peak temperature of 130 °C and duration of 4 s above 100 °C in the thermal cycle was achieved in the HAZ. It is well accepted that similar thermal cycle should be achieved in the TZ during multiple-pass FSP process. Therefore, FSP with additional water cooling only caused very weak softening in the TZ, resulting in the sound mechanical properties of the bulk FSP UFG Cu.

Clearly, the present study demonstrates that multiple-pass overlapping FSP with additional rapid cooling is an effective method of producing large-area bulk UFG materials with sound mechanical properties. It is well accepted that the FSP process is specialized in preparing sheet material, which is one of the most widely used material type in engineering application. Very large-area plates can be produced by overlapping FSP with enough passes, and the efficiency will be much enhanced by using multi-tools. Therefore, FSP should be a potential method of preparing bulk UFG materials in engineering applications.

4. Conclusions

(1) Large-area bulk UFG pure Cu was successfully prepared by multiple-pass FSP with 50% overlapping at a tool rotation rate of 400 r/min and a traverse speed of 50 mm/min under additional water cooling.
(2) Overlapping FSP did not exert a significant coarsening effect on the microstructure of the transitional zone, where similar average grain size was achieved to that of the NZ. Compared to the NZ, the transitional zone exhibited a strong {111}⟨112⟩ type A fiber shear texture.
(3) Very weak softening effect occurred in the transitional zones, and the large-area bulk UFG Cu exhibited a relatively uniform hardness distribution. Slightly reduced YS of ∼310 MPa and same UTS of ∼340 MPa and UE of ∼13% were achieved in the bulk UFG sample compared to the single-pass FSP sample.

Acknowledgments

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